Abstract No. cele502

Investigations into a Titanosilicate and Lithosilicate RUB-32

A. Celestian, A. Tripathi, J. Parise, and G. Wu (SUNY, Buffalo)

Beamline(s): X3A1

Introduction: Single crystal diffraction on micro-crystals of a titanosilicate and a lithosilicate RUB-32 (where lithium and silicon tetrahedra compose the framework) were to be solved with 2x2x5 micron and a 60x3x3 micron crystals respectively. Low synthesis yield of the titanosilicate, with very small crystals necessitated the use of synchrotron x-rays. RUB-32 was identified by powder diffraction and is a lithosilicate that forms simultaneously with RUB-29 [4], another lithosilicate. Using synchrotron radiation on these micro-crystals, it is hoped that there structures will be solved. These two materials or of interest because of their molecular sieve properties and potential catalysis properties.

Methods and Materials: Single crystal diffraction was carried out with on a Huber goniometer on the X3A1 beamline with a monochrometer tuned to .643 angstroms. Helium gas was passed through the collimator to reduce the amount of scattering by air. Data was collected on a Bruker CCD at a distance of 4.3 cm from the crystal. Each frame was exposed for 25 sec. for a full 360 degree phi scan.

Results: The structure of the titanosilicate (7.486 7.486 27.405 90.0 90.0 90.0) in space group I4(1)/amd, was solved with 4991 unique reflections and R1=.0764 and was solved from a 2x2x5 micron crystal. This structure was already found to be ETS-10 from single crystal x-ray diffraction by Wang et. al. [1] (see figure 1). The structure of RUB-32 could not be solved. RUB-32 is a complex lithosilicate where lithium is found in framework and extra frame sites. With such a small fibrous crystal (60x3x3 microns) the peak to background ratio was too long and most diffracted intensities could not be measured. SAINT [2]was used to integrated all collected data and SHELXTL [3] was used to solved the structure.

Acknowledgments: This work was supported through a grant from the National Science Foundation #DMR-0095633.

References: [1] Xiqu Wang and Allan J, Jacobson, "Crystal structure of the microporous titanosilicate ETS-10 refined from single crystal X-ray diffraction data," *J. Chem Soc. Chem. Commun.*, 973, 1999
[2] SAINT: Program to integrate and Reduce Raw Crystallographic Area Detector Data; Bruker AXS, Inc.: Madison, WI, 1996.
[3] Sheldrick, G. M. SHELXTL; Bruker AXS Inc.: Madison, WI, 1997.
[4] SH Park, JB Parise, H Gies, HM Liu, GP Grey and BH Toby, “A new porous lithosilicate with a high ionic conductivity and ion-exchange capacity,” J. Am. Chem. Soc., 122, 2000

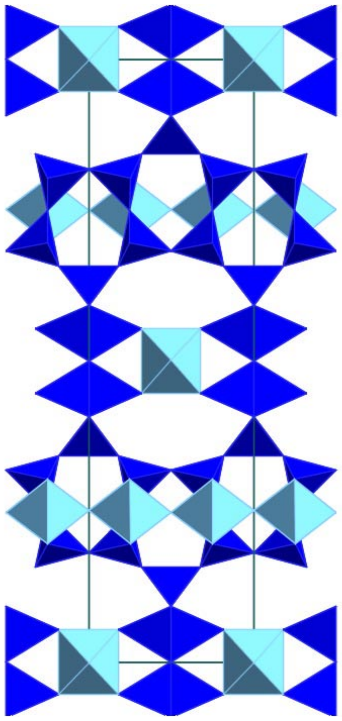

Figure 1: ETS-10 ($Na_2TiSi_5O_{13}$) viewed down (010)